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Collector Chemistry in Flotation Deinking of Xerographic Papers

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ABSTRACT

The surface hydrophobicity and the particle size of ink suspension in pulp are two important factors influencing the efficiency of flotation deinking. The main indication of hydrophobicity of ink particles is the contact angle. The contact angle of fused toner film in various surfactant and collector solutions, including TX-100, cationic surfactants, sodium oleic acid/calcium ions, and kerosene, was studied. The effect of surfactant and collector concentrations on the toner particle size was examined. The results show that the contact angle of toner film in an aqueous solution decreases as TX-100 concentration increases regardless if there is a collector. Although toner particles were hydrophobic in nature, the flotation efficiency of xerographic wastepaper can be further improved by adding certain collectors. It was found that the toner removal has a direct relationship to its surface hydrophobicity and the particle size, but other effects, such as foam stability, will also significantly affect the toner flotation. The experiments indicated that the cationic surfactant can be used as both a frothing agent and a collector for flotation deinking of toner printed wastepapers. Because a high contact angle and an optimum particle size could be obtained by adding a cationic surfactant, the best flotation deinking of xerographic paper was obtained in cationic surfactant systems.

KEYWORDS:

Flotation, Deinking, Collector, Surfactant, Toner, Contact Angle, Particle Size

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INTRODUCTION

The recycling of wastepaper is of growing importance due to the shortage of fiber supply and the restricted government regulations on solid wastepaper landfills. Although the paper recycling rate has increased steadily, the quality and cost of recycled fibers are still incompatible with virgin fibers.

Flotation deinking is one of the most important separation techniques widely used in paper recycling industry. Flotation deinking involves three main processes: detachment of the ink particles from wastepaper fibers, effective adhesion of the ink particles onto air bubble surfaces, and the removal of ink particles with the froth from flotation cells [1-3]. It has been well known that the hydrophobicity and the particle size of ink particles in flotation slurry are two of the most important factors affecting the ink-air bubble interaction. From a surface chemistry point of view, the higher the hydrophobicity of particle surface, the easier it is to remove ink particles from pulp slurry under similar flotation conditions. It is very common to apply a collector in the pulp slurry to improve the hydrophobicity of floated particles in mineral flotation. Many collectors have been successfully used in ore flotation. The most common collector used in flotation deinking is fatty acid in the presence of calcium chloride. Many researchers have focused on the understanding of ink removal by adding fatty acid/calcium collector, but the mechanism involved in this system have not been well understood [2]. Although the fatty acid/calcium collector has been traditionally used for flotation deinking of old newsprint and old magazines, Dorris and Page [4] recently indicated that fatty acid/calcium collector can also improve the toner removal efficiency during the flotation. They suggested that the increased hydrophobicity of toner particles due to the adsorption of fatty acid/calcium complex onto toner surface is the main reason for toner removal improvement. However, this hypothesis has not been validated by experimental results. Although fatty acid plus calcium chloride is an effective collector for some grades of wastepaper, the deposition of calcium fatty acid particles on the flotation equipment and paper machine is a serious problem. Therefore, it will be beneficial to the paper industry if more effective collectors that do not have side effects on the papermaking process can be developed.

It was found that the deinking of mixed office paper by flotation is more difficult than other wastepapers because a) toner is usually fused into the pores of paper and strongly adhered to the fiber surfaces, resulting in a poor toner-fiber separation during repulping [5,6], and b) the broad particle size distribution and the disk-like shape of toner particles results in a poor attachment between toner particles and air bubbles [7, 8]. Based on these understandings it is believed that the toner particles must be kept hydrophobic in flotation cell and the optimum particle size must be achieved to improve the flotation deinking efficiency of toner printed papers.

This study is to develop a fundamental understanding of collector chemistry in flotation deinking of toner printed papers. The relationship between the toner surface chemistry, the agglomeration of toner particles in solutions, and flotation efficiency was investigated.

EXPERIMENTAL

Chemicals

Triton X-100 (analyze grade, J. T. Backer Inc.), cetyltrimethylammonium bromide [CTMAB, Aldrich, 95%], dodecyltrimethylammonium bromide [DTMAB, Aldrich, 99%], kerosene (commercial product), sodium oleic acid sodium salt [Aldrich, 98%], and calcium chloride [Aldrich, tech.] were used as received.

Contact Angle Measurements

The surface tension of liquid was measured by a dynamic contact angle analyzer (Cahn DCA 312) using a glass plate. Toner samples for contact angle measurement were made by copying 3 layers of Xerox toner on both sides of a transparency. The coated transparency was cut into 20mm x 30mm pieces before use. The wetting force of coated toner film in different solutions was measured, and the contact angle was calculated using Wilhelmy principle. Both surface tension and contact angle measurements were repeated several times until a stable reading was obtained.

Particle Size Measurements

The particle size and size distributions of toner suspension in different solutions were measured by a laser diffraction-based particle size analyzer (Malvern 2600, Malvern Instruments, MA). The system consists of 2mW He-Ne Laser (633nm wavelength), a Transmitter with 9mm beam expansion, a Receiver with a Fourier transform lens, and a 31-element solid state detector array in concentric semicircular annuli design. The sample used for particle size analysis were made by dispersing Xerox toner in a pulp filtrate in the presence or absence of surfactant.

Flotation Deinking

The pulp was made from bond papers printed by Xerox toner with a fixed pattern of X. The papers were pulped at pH of 9.3 and a consistency of 10.5% without adding any chemicals except sodium hydroxide. A laboratory flotation cell was used to conduct the flotation deinking. The deinking cell was made from a polyacrylate pipe with a height of 80cm and a diameter of 10cm. Nitrogen was blown into the pulp suspension at a rate of 14 ± 0.075 SLPM (standard liter per minute) through an air filter (pore size $\sim 50 \mu\text{m}$) at the bottom of the flotation cell. The air flow rate was measured by an Omega FMA1700/1800 flowmeter. The consistency of the pulp used in flotation was 0.5%. The flotation time was 10 minutes for all experiments.

The handsheets for brightness analysis were made on a 15-cm Büchner funnel according to TAPPI standard method T218 om-91. The brightness of a handsheet was measured by a UV-VIS spectrophotometer (Shimadzu UV-160A) using TAPPI Standard method T452 om-92.

RESULTS AND DISCUSSION

Calcium Fatty Acid as a Collector in Toner Flotation Deinking

In the flotation deinking of mixed office wastepapers, a nonionic surfactant has been commonly used as a dispersant and frothing agent. However, the addition of a nonionic surfactant, such as TX-100, will reduce the hydrophobicity of toner particles, therefore the

toner removal efficiency by flotation will be decreased. The reduction of toner particle hydrophobicity due to adsorbed surfactant can be clearly seen from Fig. 1, i.e., the advancing contact angle of aqueous solution on a toner film decreases as the concentration of TX-100 increases. When the concentration of TX-100 increases from 0 to 80mg/L (the typical concentration for mixed office waste deinking in industry practice), the advancing contact angle decreases from 140 to 75 degrees. With a further increase in concentration to 200mg/L, the advancing contact angle decreases to 10 degree or even lower. The significant decrease in the contact angle must result in a reduction in deinking efficiency as will be discussed later.

Because nonionic surfactant usually leads to a reduction in the contact angle of toner particles in pulp suspension, it is interesting if there are some collectors that can restore the contact angle of toner particles. It has been known that calcium fatty acid can be used as a collector for both old newsprint and mixed office waste papers [4,9,10]. Therefore, the collector chemistry of calcium fatty acid was first examined in this study.

The contact angle of toner film in water as a function of sodium oleic acid concentration in the presence of 500mg/L calcium chloride is also shown in Fig. 1. It can be seen that as the concentration of sodium oleic acid increases, the advancing contact angle decreases rather than increases. This suggests that although the complex formed by sodium oleic acid and calcium chloride is hydrophobic, the hydrophobicity of toner surface will not be improved by adsorption of this complex because the hydrophobicity of toner itself is higher than that of calcium fatty acid aggregates.

To effectively remove toner particles from pulp slurry, a relatively stable foam is essential. The common method to generate a foam layer is to add frothing agent directly into pulp suspension during stock preparation. Therefore, it is very interesting to know how the interaction between calcium fatty acid and a frothing agent will affect the hydrophobicity of toner particles in a pulp system. Fig. 1. Shows that in the presence of 50 mg/L sodium oleic acid and 250 mg/L calcium chloride, the toner remained at a high contact angle (>105 degrees) until the concentration of TX-100 was increased up to 130 mg/L. Comparing that with the contact angle obtained in the absence of sodium oleic

acid/calcium ions (curve A in Fig. 1), it can be seen that calcium fatty acid can protect the toner surface from the reduction of the hydrophobicity due to the adsorption of TX-100. This effect is more significant at low TX-100 concentrations (< 130 mg/L). When TX-100 concentration is higher than 130 mg/L, the advancing contact angle of toner film suddenly decreased from 105 to 45 degrees. Although the reason for this sharp decrease is not clear, the solubilization of fatty acid by TX-100 at the concentration close to TX-100's critical micellization concentration (185 mg/L) may be one of the possible effects.

Although the hydrophobicity of ink particles is one of the dominating factors in flotation deinking, the particle size is also critical. Obviously, an effective collector should not only remain a high contact angle of ink particles in solution, but also agglomerate the ink particles into an optimum size. Fig. 2 shows the particle size in aqueous solutions as a function of the concentration of TX-100 or sodium oleic acid. It has been noted from toner particle size measurements that the number distribution of toner particle the presence of TX-100 has normal Gauss distribution, regardless of the concentration of TX-100. It can be seen that, when TX-100 was used alone, the toner particle size slightly decreased as the concentration of TX-100 was increased. This is not surprising because although the toner particles can be dispersed in the pulp filtrate and stabilized by adsorbed anionic trash (they aggregate and floated in pure water), there must be some small toner aggregates because anionic trash in the pulp filtrate is not a very effective stabilizer. When TX-100 was added in the suspension, these small toner aggregates were redispersed by surfactant resulting in a decrease in the average particle size. However, it can also be seen from Fig. 2 that in the presence of 500 mg/L calcium chloride and 20 mg/L TX-100, the particle size of toner suspension increases as the concentration of sodium oleic acid increases. This suggests that toner particles were aggregated by calcium oleic acid even in the presence 20 mg/L TX-100. At 90 mg/L of sodium oleic acid solution, the mean toner particle size of toner powders increased to 27 μm (Sauter Mean Diameter), which was in the optimum size range for flotation reported by Ferguson [2]. It was noted that the increase of collector concentration will not only increase the number average particle size, but also the particle size distribution. For example, it is found that the agglomeration of small particles was more significant than that of large particles.

The effect of hydrophobicity and particle size of toner in pulp slurry on the flotation deinking efficiency was investigated by measuring the brightness gain of the handsheets made from deinked xerographic fibers. The brightness gain as a function of TX-100 in either the presence or absence of calcium oleic acid is shown in Fig. 3. It can be seen that in the absence of calcium oleic acid, the brightness gain increases as TX-100 concentration increases up to 120 mg/L, then decreases as TX-100 concentration further increases. The initial increase in the brightness at low TX-100 concentration is due to the increase in the foam stability, and the decrease in the brightness at high concentration is due to the decrease in the hydrophobicity of toner particles by adsorbing TX-100 molecules onto the surface, which is consistent with the contact angle measurement shown in Fig. 1. This behavior has also been reported in our previous study [11] and in Eppler et al. [12].

It is reasonable to assume that toner removal can be enhanced by adding a fatty acid and calcium ions because the toners have a high contact angle in the solution, as has been reported previously [4]. Surprisingly, our results shown in Fig. 3 indicate that the presence of 50 mg/L sodium oleic acid and 250 mg/L calcium chloride decreases rather than increases the toner removal in similar pulp systems. Although the mechanism of this reduction in flotation deinking efficiency is not clear, the effect of calcium fatty acid on the foam stability and structure must be accounted. It was observed during the flotation experiments that the foam was much less stable in the presence of calcium fatty acid compared with that using TX-100 alone. Because the bubbles were broken during their rise to the top of the flotation cell, some adhered toner particles returned back to the pulp slurry, which may result in a decrease in the flotation deinking efficiency.

Kerosene as a Collector in Toner Flotation Deinking

Hydrocarbon materials that have been widely used in the mineral flotation industry as collectors are getting attention in the paper industry. Snyder and Berg [7] described that some hydrocarbon materials can be used as an agglomeration agent for suspended toner particles. Pelton [13] also found that ink particles are more easily adhered to hydrocarbon oil coated glass beads. Miller et al. [14] found that the toner flotation efficiency can be significantly improved by adding 5% tetrahydrofuran or acetone (based on dry waste) into

pulp suspension. In a recent study [15], Oguz indicated that mixture of kerosene, detergent, and borax can significantly improve ink removal. Although these findings are interesting, no fundamental study on the collector chemistry using a mineral oil or hydrocarbon material in flotation deinking has been reported in the literature. In this study, the kerosene was used as a model hydrocarbon collector for toner deinking. The contact angle of toner film in water was measured and the results are shown in Fig. 4. It can be seen that although the advancing contact angle of toner decreases slightly as kerosene concentration is increased, it is still as high as 130 degree at kerosene concentrations of up to 160 mg/L (small kerosene droplets on the surface of the solution will be seen with further increases in concentration). However, when TX-100 was added into toner suspension in the presence of 50 mg/L kerosene, the advancing contact angle was decreased. Although this decrease is significant, by comparing with curve C in Fig. 4, it can be seen that the addition of kerosene can restore some of the loss in toner hydrophobicity that is caused by the adsorption of TX-100.

The effect of kerosene on the agglomeration of toner particles in the presence of TX-100 was also examined and the results are shown in Fig. 5. It can be seen that the particle size of toner in the presence of 20 mg/L TX-100 increases significantly as the increase in the kerosene concentration, suggesting a significant agglomeration of toner particles in this solution. It is believed that the agglomeration of toner particles was caused by “oil bridging” mechanism as reported by Berg et al. [7] using other hydrocarbon solvents.

The effect of kerosene on the flotation deinking efficiency of toner printed papers was studied using TX-100 as a frothing agent. The brightness gain of the handsheets made of recycled fibers is shown in Fig. 6. It can be seen that in the presence of 20 mg/L kerosene, the ink removal is higher than that of without kerosene, particularly at low TX-100 concentration. This is consistent with the contact angle and particle size measurement, i.e., a high contact angle and an optimum particle size result in a high deinking efficiency. The results shown in Fig. 6 indicate that kerosene can be used as a collector for toner flotation deinking when TX-100 is used as a frothing agent.

Toner Flotation Deinking Using Cationic Surfactants

Cationic surfactants have been traditionally used in mineral flotation. One of the advantages of using cationic surfactants is that these chemicals can function as both a collector and a frothing agent for some ores. The effect of cationic surfactant structure on the mineral flotation was studied [16-18]. Although cationic surfactants are very effective for flotation of some mineral particles, limited research [1,2,19,20] has been done using cationic surfactants for flotation deinking. There is no report in the literature on the application of cationic surfactants to deink toner-printed wastepaper.

Fig. 7 shows the advancing contact angle of toner film in different cationic surfactant solutions. It can be seen that the contact angles of toner film obtained in two different cationic surfactants are much higher than that in the TX-100 solution. More interesting is that the contact angle increases initially as the concentration of CTMAB increases, then decreases as further increase in CTMAB concentration. It is known that the toner particles in wood pulp suspension are negatively charged because of the adsorption of soluble anionic polymers, such as sulphonated lignin and fatty acids from wood fibers, onto the toner surfaces. When cationic surfactant molecules are adsorbed onto these negatively charged toner surfaces, the configuration of adsorbed cationic surfactant should be different from that of anionic and non anionic surfactants. At a low concentration of cationic surfactant, the positively charged surfactant heads anchor to the negatively charged toner surface leaving hydrophobic tail toward the solution. As a result, the contact angle is increased. However, at a high concentration of cationic surfactant, a double layer adsorption can occur and the hydrophilic heads of cationic surfactant orient to the water phase, which reduces the contact angle. The configurations of cationic surfactant at different concentrations are schematically shown in **Fig. 8**. It should be noted that no contact angle increase was observed for the cationic surfactant of DTMAB, which may be attributed to the fact that the DTMAB has a shorter hydrocarbon chain than the CTMAB, resulting a more hydrophobic adsorption layer on the toner surface.

The effect of cationic surfactants on the agglomeration of toner particles is shown in **Fig. 9**. The particle size of toners in pulp filtrate was almost a constant when the concentration

of DTMAB was increased, but increased steadily with the increase of CTMAB concentration up to 90 mg/L. The particle size increase is consistent with the hydrophobicity increase of toner particles in cationic surfactant solutions. It is believed that in addition to the hydrophobicity effect, the charge neutralization between cationic surfactant and negatively charge adsorbed materials on the toner surface in a pulp filtrate will also play an important role in the toner particle agglomeration.

As discussed previously, the toner particle hydrophobicity can remain relatively high and particle size can be increased in CTMAB solutions. It was also found that cationic surfactants are effective frothing agents as well as collectors in pulp suspensions. Therefore, it is not necessary to have a second surface active agent in flotation deinking if a cationic surfactant is used. This advantage may significantly reduce the flotation deinking cost. The brightness gain obtained from handsheets made of deinked fibers as a function of cationic surfactant concentration is shown in Fig. 10. It was found that cationic surfactants produced much higher brightness gains than TX-100. The result is consistent with the contact angle and particle size measurements, i.e. CTMAB and DTMAB are very effective collectors and frothing agents for toner flotation deinking.

CONCLUSIONS

- 1) The contact angle of toner film in water-surfactant solution decreases with the increase of TX-100 concentration independent of the presence of a collector. The balance between froth stability and hydrophobicity leads to an optimized concentration for toner removal if TX-100 is used alone.
- 2) The addition of fatty acid/calcium ions causes particle agglomeration but does not increase the contact angle of toner particles. The addition of sodium oleic acid/calcium ions in the flotation process does not enhance the toner removal if TX-100 is used as a frothing agent.
- 3) Kerosene can increase the toner particle size and maintain a relatively high contact angle of toner in TX-100 solution. The flotation deinking efficiency of toner-printed

papers can be improved by adding a small amount of kerosene if TX-100 is used as a frothing agent.

- 4) Cationic surfactant can be used as both a collector and a frothing agent. For the systems investigated in this study, the cationic surfactants provided the best ink removal.

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Figure 1. Advancing contact angle of toner film in aqueous solutions as a function of A: TX-100 concentration without any other chemicals; B: TX-100 concentration in the presence of 50 mg/L sodium oleic acid and 250 mg/L calcium chloride; C: sodium oleic acid concentration in the presence of 500 mg/L calcium chloride.

Figure 2. Toner particle size as a function of A: TX-100 concentration; and B: TX-100 concentration in the presence of 50 mg/L sodium oleic acid and 500 mg/L calcium chloride.

Figure 3. The brightness gain of handsheets made from deinking fibers as a function of A: TX-100 concentration without other chemicals; B: TX-100 concentration in the presence of 50 mg/L sodium oleic acid and 250 mg/L calcium chloride.

Figure 4. Advancing contact angle of toner film in aqueous solutions as a function of A: TX-100 concentration without any other chemicals; B: TX-100 concentration in the presence of 50 mg/L kerosene; C: kerosene concentration.

Figure 5. Toner particle size as a function of A: TX-100 concentration; B: kerosene in the presence of 20 mg/L TX-100.

Figure 6. The brightness gain of handsheets made from deinking fibers as a function of A: TX-100 concentration; B: TX-100 concentration in the presence of 20 mg/L kerosene.

Figure 7. Advancing contact angle of toner film in aqueous solutions as a function of A: TX-100 concentration; B: DTMAB concentration; C: CTMAB concentration.

Figure 8. The orientation of cationic surfactant on a negatively charged toner surface. A: monolayer adsorption at low concentration, and B: double layer adsorption at high concentration.

Figure 9. Toner particle size as a function of A: TX-100 concentration; B: DTMAB concentration; C: CTMAB concentration.

Figure 10. The brightness gain of handsheets made from deinking fibers as a function of A: TX-100 concentration; B: DTMAB concentration; C: CTMAB concentration.

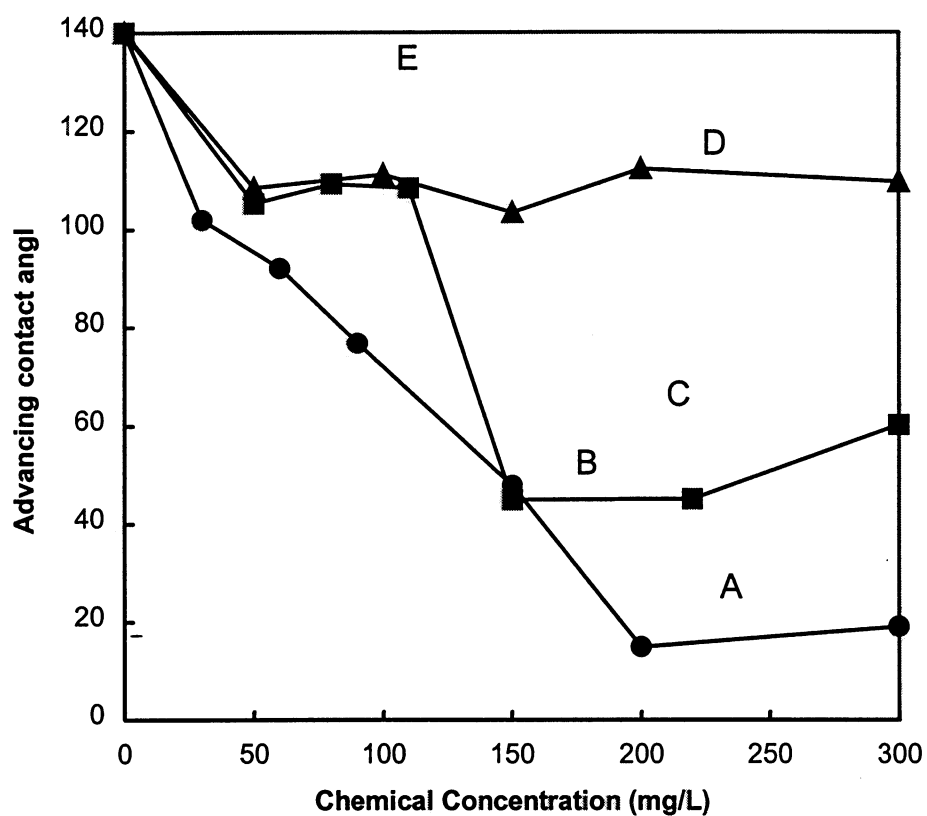


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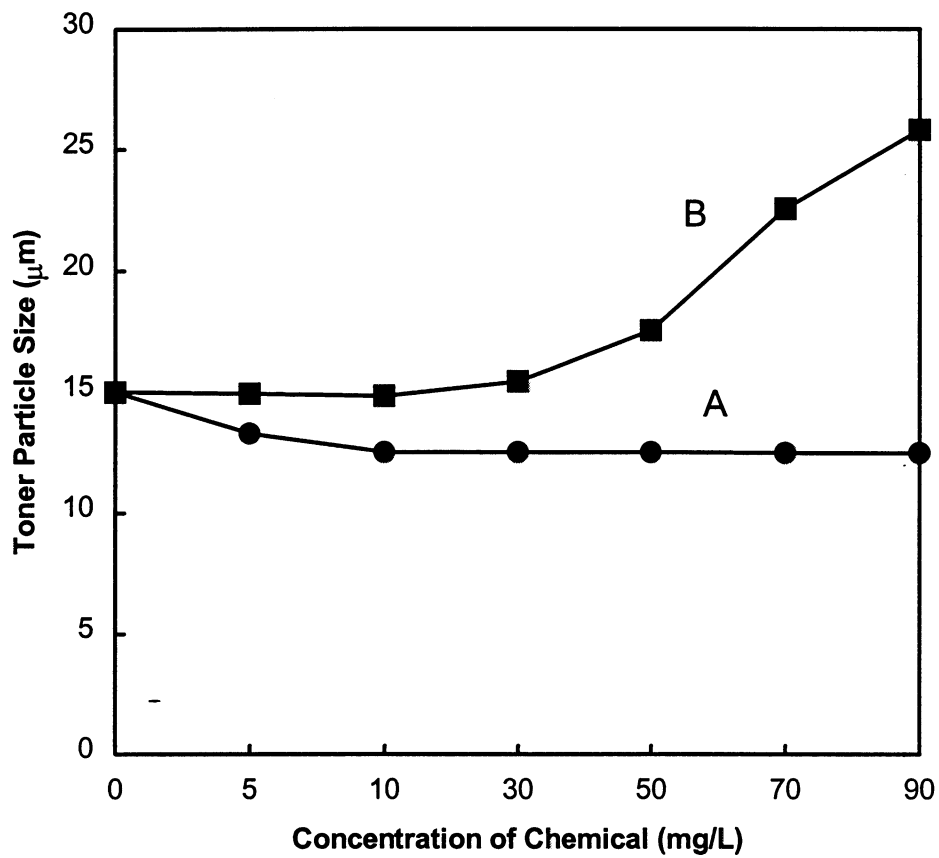


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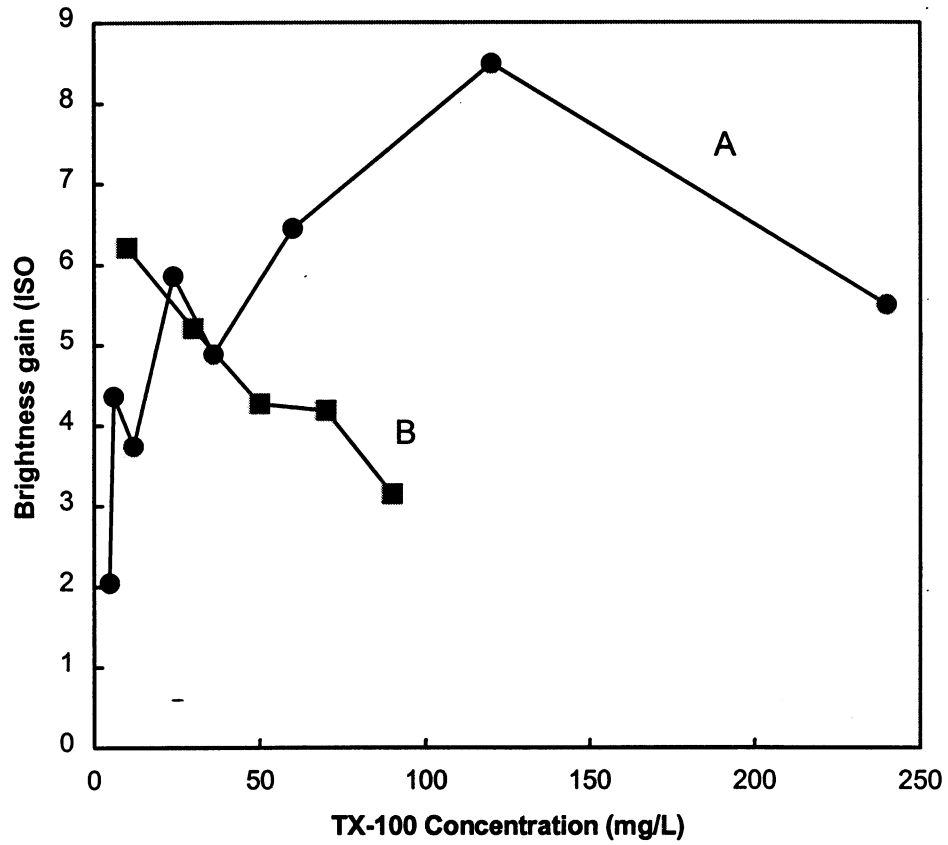


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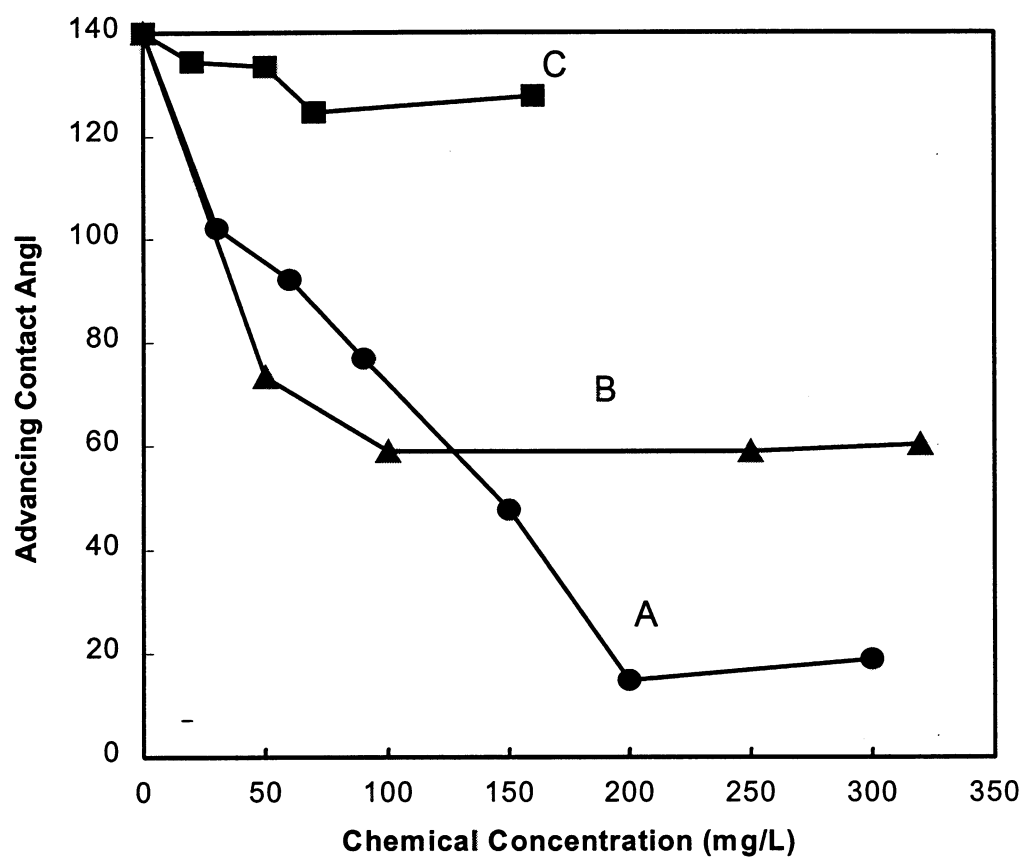


Figure 4. Advancing contact angle of toner film in aqueous solutions as a function of A: TX-100 concentration without any other chemicals; B: TX-100 concentration in the presence of 50 mg/L kerosene; C: kerosene concentration.

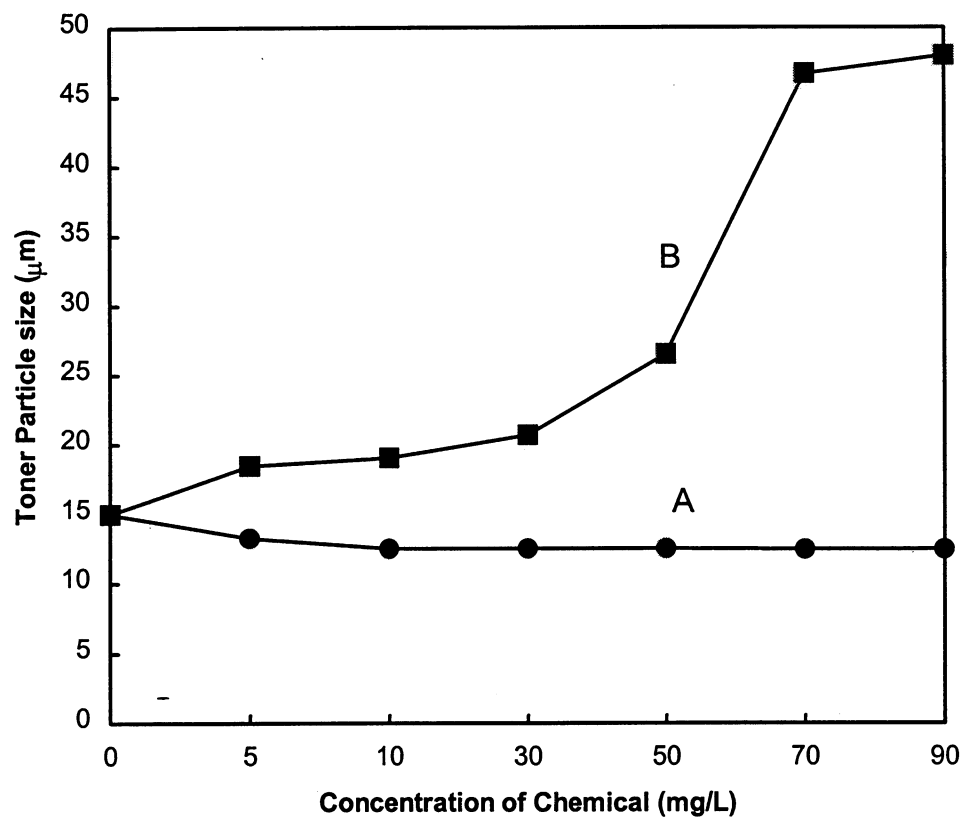


Figure 5. Toner particle size as a function of A: TX-100 concentration; B: kerosene in the presence of 20 mg/L TX-100.

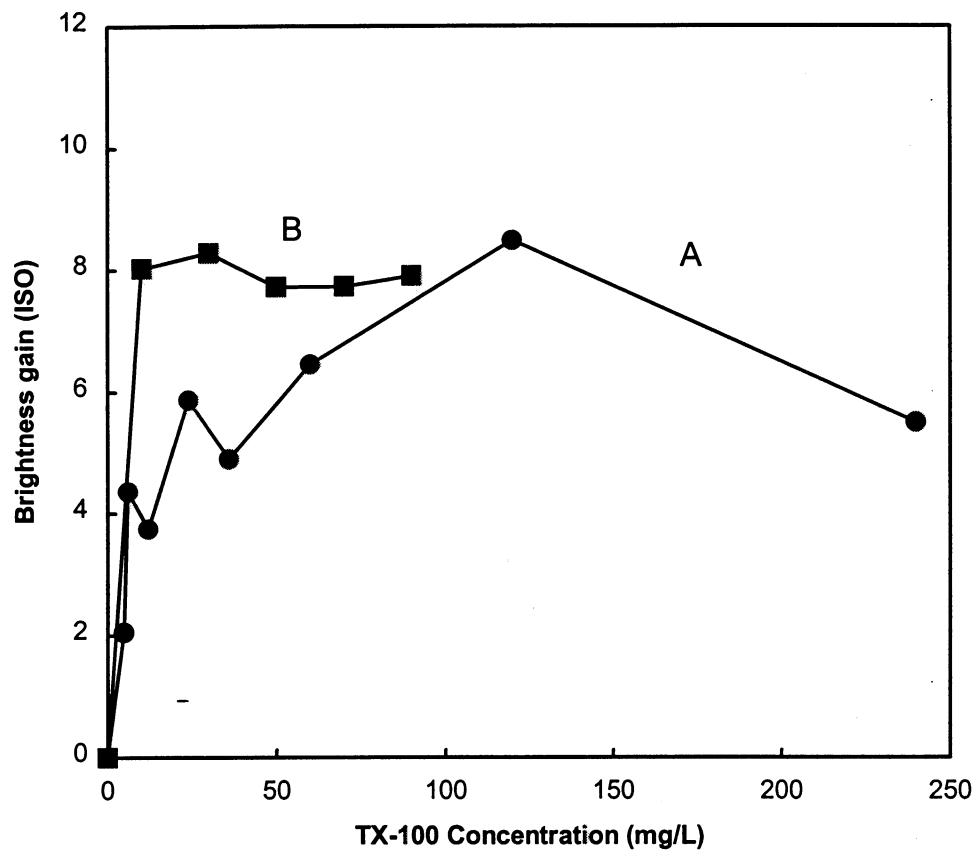


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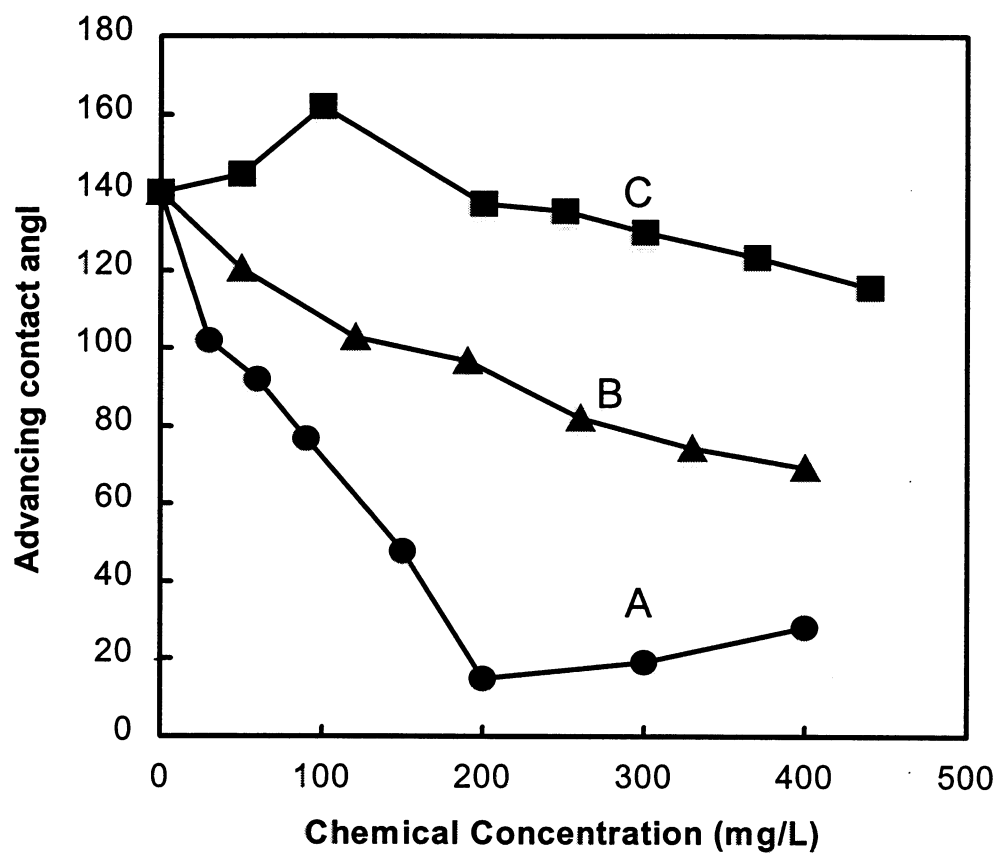


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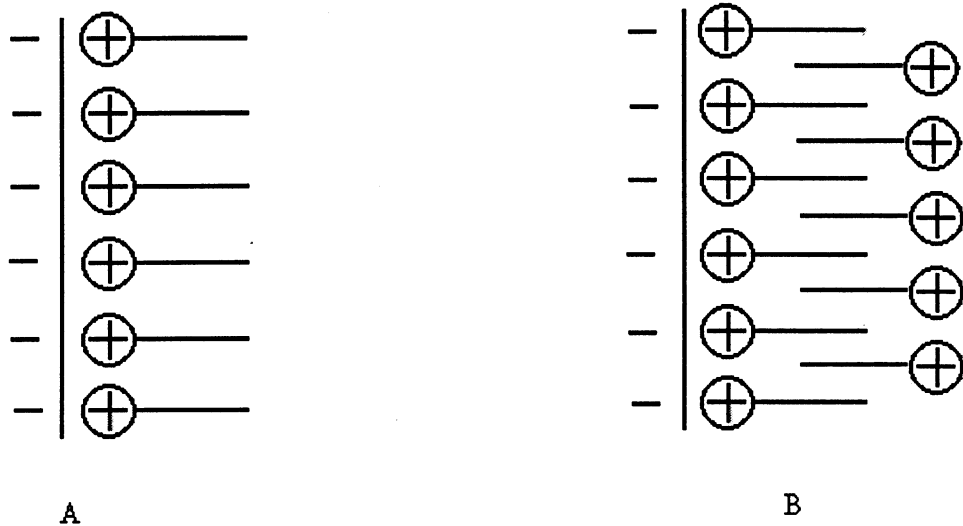


Figure 8. The orientation of cationic surfactant on a negatively charged toner surface.

A: monolayer adsorption at low concentration, and B: double layer adsorption at high concentration.

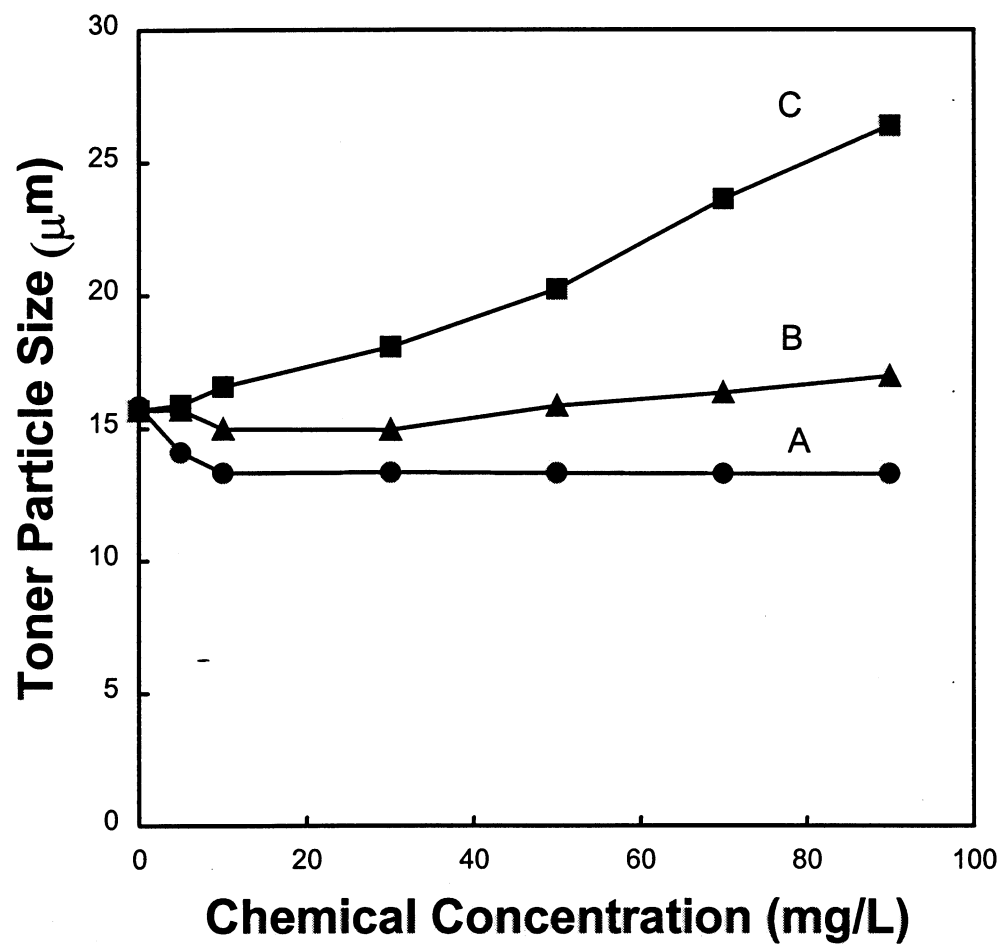


Figure 9. Toner particle size as a function of A: TX-100 concentration; B: DTMAB concentration; C: CTMAB concentration.

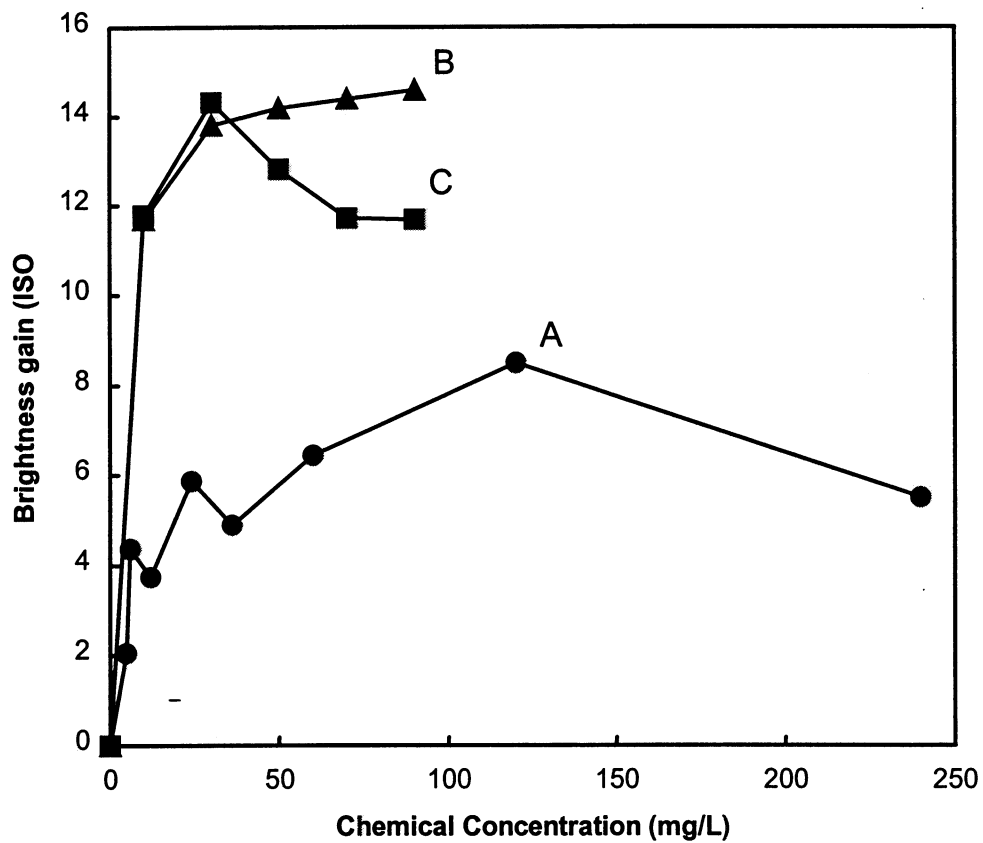


Figure 10. The brightness gain of handsheets made from deinking fibers as a function of
A: TX-100 concentration; B: DTMAB concentration; C: CTMAB concentration.

